PATENT Case 4233C3

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:

Donald B. Appleby et al

Group Art Unit: 1211

Serial No.:

08/360,184

Examiner: E. White

Filed:

December 20, 1994

For:

**Polyol Polyester Synthesis** 

## DECLARATION UNDER 37 C.F.R. §1.608 OF RICHARD G. SCHAFERMEYER

Assistant Commissioner for Patents Washington, DC 20231

Dear Sir:

I, RICHARD G. SCHAFERMEYER, declare that:

- 1. I am a co-inventor of and am familiar with the present Appleby et al U.S. patent application.
- 2. I have a Bachelor of Science degree in Chemical Engineering received from the University of Missouri Rolla in 1973, and a Master of Science degree in Chemical Engineering received from the University of Missouri Rolla in 1975.
- 3. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1975.

During my employment with The Procter & Gamble Company, I worked, inter alia, on developing a continuous process for the synthesis of polyol fatty acid esters, specifically sucrose fatty acid esters, referred to herein as sucrose polyesters, FG or FG base, by reaction of sucrose and fatty acid methyl ester.

- 4. From about 1987 to the present, I have worked first as a Section Head and later as an Associate Director on the sucrose polyester production project. My responsibilities included supervising process and product development, studying process parameters, reviewing data, planning pilot plant runs, and coordinating and organizing information and activities relating thereto.
- 5. During my employment with The Procter & Gamble Company, I have, from time-to-time, prepared and distributed bi-weekly and monthly reports accurately describing my activities, and/or activities of those under my direction and control, during the prior two week or monthly period which was the subject of the report. For each report, I accurately set forth therein the ending date for the prior two week or monthly period which was the subject of the report. I have examined Exhibits 54-58, and I confirm that each of these exhibits comprises an accurate copy of a bi-weekly or monthly report which I authored and which bears my signature.
- 6. I have reviewed the Declaration Under 37 C.F.R. § 1.608 of Ephraim L. Kelly and Exhibit No. 35 identified in the Kelly Declaration, both of which Declaration and Exhibit are submitted herewith. The Kelly Declaration accurately describes experiments conducted by Mr.

Kelly under my general direction and control for studying and developing the sucrose polyester process and accurately describes the procedures for recording the general nature and results of such experiments as respectively described therein.

- 7. I have reviewed Exhibit 54 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report, which I prepared on or about February 22, 1989. As used in Exhibit 54, "soap" refers to the soap used as a emulsifier. As set forth in Exhibit 54, potassium hydroxide (KOH) is a stronger base than the potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) which had generally been used as the reaction catalyst, and I suggested at that time that residual potassium hydroxide in the soap could act as a catalyst for the sucrose polyester synthesis reaction.
- 8. As accurately described in the Kelly Declaration, ¶¶5-8, and Exhibit 35, a matrix of experiments was performed by Mr. Kelly under my general direction and control in March and April of 1989 to investigate the effect of using residual potassium hydroxide in the potassium soap emulsifier as the only catalyst in the reaction of sucrose and fatty acid methyl ester (i.e., potassium carbonate was not added to the reaction), as disclosed at page 14, lines 15-27 of the Appleby et al application. The experiments demonstrated that sucrose ester product having high octaester content and I-bar values (the average degree of esterification based on mole fractions) greater than about 7.5 were obtained (Kelly Declaration, ¶¶6-8) using the residual potassium hydroxide as the sucrose polyester reaction catalyst.

- 9. I have reviewed Exhibit 55 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report, which I prepared on or about January 25, 1989. Although the sucrose polyester synthesis is the dominating reaction, minor side reactions, such as difatty ketone (DFK) formation, can occur during the synthesis of sucrose polyester. As set forth in Exhibit 55, DFK formation was considered a competitive reaction to the sucrose esterification reaction, and most of the DFK which formed during sucrose polyester synthesis formed during the subsequent (i.e. non-initial) stages of the reaction. As further set forth in Exhibit 55, a project was underway to study and reduce the DFK formation in the sucrose polyester reaction. As disclosed at page 24, lines 1-16 of the Appleby et al application, low levels of DFK in the sucrose polyester product are preferred.
- 10. I have reviewed Exhibit 56 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Biweekly Report, which I prepared on or about June 28, 1989. Exhibit 56 describes the recent progress at that time of the DFK investigation. As set forth in Exhibit 56, sucrose polyesters having octaester contents of about 78-88% and difatty ketone levels of about 220-300 ppm were recently produced, while sucrose polyester having octaester content of about 79% and 70 ppm DFK had been produced using a lower temperature in a subsequent reaction stage. The Appleby et al application similarly discloses at page 18, lines 6-10 and page 24, lines 1-5 that low temperatures are preferred for reducing the level of DFK in the polyol polyester product.

- 11. I have reviewed Exhibit 57 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Division Biweekly Report, which I prepared on or about September 6, 1989. Exhibit 57 describes the recent progress at that time of the DFK investigation. As set forth in Exhibit 57, a termination theory was proposed wherein it was believed that a reduction in the available hydroxyl groups as the reaction proceeds increases formation of the DFK precursor. It was also theorized that the activation energy of the sucrose polyester reaction appeared to be different than the Claisen condensation reaction involved in the formation of difatty ketones. As additionally set forth in Exhibit 57, it was again observed that lower temperatures in subsequent reaction stages appeared to reduce DFK formation.
- 12. I have reviewed Exhibit 58 and confirm that it is an accurate copy of the Industrial Chemicals Product Development Division Food Ingredients Monthly Report, which I prepared on or about October 1, 1989. Exhibit 58 describes the recent progress at that time of the DFK investigation. As set forth in Exhibit 58, a lower subsequent stage temperature (about 100°C, or about 212°F) allows the reaction to attain high octaester levels (from about 82% to about 84%, by weight of total sucrose polyester) while at the same time keeping the DFK levels below about 300 ppm.
- 13. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States

Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

RICHARD G. SCHAFERMEYER

Date: 4-1-99

0368409.01